IS 718 (1977): Carbon Tetrachloride [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

BLANK PAGE

IS : 718 - 1977
(Reaffirmed 1995)

Indian Standard

SPECIFICATON FOR CARBON TETRACHLORIDE

(Second Revision)

Third Reprint SEPTEMBER 1998

UDC 661.723.24

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7

February 1978

AMENDMENT NO. 1 JANUARY 1987

TO

IS:718-1977 SPECIFICATION FOR CARBON TETRACHLORIDE

(Second Revision)

[Page 5, Table 1, col 2, Sl No. (vii)] - Substitute 'Carbon disulphide, percent by mass, Max' for 'Sulphur compounds (as CS_2), percent by mass, Max'.

(Page 16, clause A-8, heading) - Substitute the following for the existing matter:

'A-8. DETERMINATION OF CARBON DISULPHIDE'

(PCDC 9)

Printed at New India Printing Press, Khurja, India

AMENDMENT NO. 2 SEPTEMBER 1988
TO
IS : 718 - 1977 SPECIFICATION FOR CARBON TETRACHLORIDE

(Second Revision)

[*Page* 5, *Table* 1, *col* 3, *Sl No.* (ii)] — Substitute the following for the existing:

'The difference between the temperature (running points) at which 2 and 97 percent of the volume taken have been collected shall not exceed 1°C and the range shall include the temperature of 76·7°C.'

[*Page* 5, *Table* 1, *col* 4, *Sl No.* (ii)] — Substitute the following for the existing:

'The difference between the temperature (running points) at which 2 and 97 percent of the volume taken have been collected shall not exceed 1·4°C and the range shall include the temperature of 76·7°C'

(PCDC 9)

Printed at New India Printing Press, Khurja, India

Indian Standard

SPECIFICATON FOR CARBON TETRACHLORIDE

(Second Revision)

Organic Chemicals (Miscellaneous) Sectional Committee, PCDC 9

Chairman	*Representing*
SHRI S. L. VENKITESWARAN	Amar Dye-Chem Ltd, Bombay
Members	
DR K. AGHORAMURTHY	Indian Petro-Chemicals Corporation Ltd, Vadodara
DR K. C. SINGHAL (*Alternate*)	
SHRI P. V. ARUR	Hindustan Organic Chemicals Ltd, Rasayani
SHRI A. K. BHATTACHARYA	National Test House, Calcutta
DR D. K. DAS (*Alternate*)	
DR S. P. BHATTACHARYA	Directorate General of Technical Development, New Delhi
DR I. P. BUCH	Hindustan Antibiotics Ltd, Pune
SHRI S. K. CHAKRABORTY	Development Commissioner (Small Scale Industries), New Delhi
DR R. B. CHAUDHURY	Carbide Chemicals Ltd (Division of Union Carbide India Ltd), Bombay
SHRI V. D. JOSEPH (*Alternate*)	
DR S. K. DATTA	National Organic Chemical Industries Ltd, Bombay
SHRI S. S. AHLUWALIA (*Alternate*)	
DR S. S. GOTHOSKAR	Directorate General of Health Services, New Delhi
SHRI R. BALASUBRAMANYAN (*Alternate*)	
DR HARI SINGH	Ministry of Defence (R & D)
SHRI N. G. S. IYER	Directorate General of Supplies & Disposals, New Delhi
SHRI G. C. MOTWANI	Indian Chemical Manufacturers' Association, Calcutta
SHRI M. S. RAMAMURTHY (*Alternate*)	
DR C. R. SAHU	Durgapur Chemicals Ltd, Durgapur
DR H. S. SARKAR (*Alternate*)	
SHRI SANTOKH SINGH	National Chemical Industries, New Delhi
SHRI BRIJINDER PAL SINGH (*Alternate*)	
SHRI N. N. SHANBHAG	Herdillia Chemicals Ltd, Bombay
SHRI I. FERNANDES (*Alternate*)	

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
DR D. R. SHRIDHAR	Indian Drugs and Pharmaceuticals Ltd, New Delhi
SHRI M. R. TYAGARAJAN	S. T. C. of India Ltd, New Delhi
DR R. V. VENKATARATNAM	Regional Research Laboratory (CSIR), Hyderabad
DR N. K. SOGANI (*Alternate*)	
DR G. M. SAXENA, Director (Chem)	Director General, BIS (*Ex-officio Member*)

Secretary

SHRI S. C. KALRA
Assistant Director (Chem), BIS

Chloroderivatives Subcommittee, PCDC 9 : 4

Convener

SHRI M. PADMANABAN	The Mettur Chemicals and Industrial Corporation Ltd, Mettur Dam

Members

SHRI D. SESHASAYEE (*Alternate* to Shri M. Padmanaban)	
SHRI R. C. BHATTACHARYA	Directorate General of Technical Development, New Delhi
SHRI R. C. SHARMA (*Alternate*)	
DR S. K. DATTA	National Organic Chemical Industries Ltd, Bombay
SHRI S. S. AHLUWALIA (*Alternate*)	
SHRI V. S. MANKIKAR	Ahmedabad Mfg & Calico Printing Co Ltd, Bombay
DR B. R. C. ANAND (*Alternate*)	
DR S. N. NAG	Hindustan Insecticides Ltd, New Delhi
SHRI T. V. RAMACHANDRAN	Dhrangadhra Chemical Works Ltd, Bombay
SHRI V. RAMADURAI	The Standard Mills Co Ltd, Bombay
SHRI A. SWAMINATHAN (*Alternate*)	
DR G. RAMANA RAO	Indian Drugs & Pharmaceuticals Ltd, Hyderabad
DR S. N. MAHAJAN (*Alternate*)	
DR C. R. SAHU	Durgapur Chemicals Ltd, Durgapur
SHRI A. K. SENGUPTA (*Alternate*)	
SHRI H. K. VENKATARAMAIH	Hindustan Organic Chemicals Ltd, Bombay
SHRI A. D. PATANKAR (*Alternate*)	
SHRI H. A. VORA	Vijay Industries, Bombay
SHRI S. BALASUBRAMANIAM (*Alternate*)	

Indian Standard

SPECIFICATION FOR CARBON TETRACHLORIDE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 5 August 1977, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1955 and subsequently revised in 1970 prescribing two grades of the material and including the requirement for methyl chloride, methylene chloride and chloroform content. In the present revision, the requirements for relative density and distillation range for both the grades have been modified. For Grade 1 the non-volatile matter content has also been changed. The colour of the material has been given in Hazen units and the method of test included. Further, operating parameters using hydrogen as carrier gas have also been included. The various methods of test have been modified suitably.

0.3 Carbon tetrachloride is used extensively as a solvent and diluent. It is also used for the manufacture of refrigerants of chlorofluoro methane series, in grain fumigants, in fire extinguishing blends and for moth control purposes.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for carbon tetrachloride (CCl_4).

*Rules for rounding off numerical values (*revised*).

2. GRADES

2.1 The material shall be of the following grades:

a) *Pure* — generally used for the manufacture of refrigerants of chlorofluoro methane series.

b) *Technical* — for industrial purposes.

3. REQUIREMENTS

3.1 Description — The material shall consist essentially of carbon tetrachloride (CCl_4), and shall be a clear liquid free from sediment and suspended matter.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of the Appendix is given in col 5 of the table.

4. PRECAUTIONS IN HANDLING

4.1 Widespread application of carbon tetrachloride often without evidence of ill-effects has resulted in underrating its toxicity. Careful investigations have shown carbon tetrachloride to be one of the most harmful of the common solvents. Extreme care, therefore, shall be taken in handling it (*see* **5.2.1**, **5.2.2** and IS : 5311-1969*).

5. PACKING AND MARKING

5.1 Packing — The material shall be packed as agreed to between the purchaser and the supplier. It is recommended that, as a finished product, carbon tetrachloride, be handled in galvanized or epoxy-lined mild steel containers rather than plain steel and cast iron drums.

5.1.1 Each container shall be securely closed, preferably with replaceable closures.

5.2 Marking — The containers shall be marked with the name and grade of the material; net mass; manufacturer's name and recognized trade-mark, if any; and the date, month and year of manufacture.

5.2.1 All containers in which the material is stored or transported shall be prominently and clearly marked with the following:

'VOLATILE, POISONOUS SOLVENT. USE WITH ADEQUATE VENTILATION. AVOID PROLONGED BREATHING OF VAPOUR. AVOID CONTACT WITH FLAME OR HOT SURFACE.'

*Code of safety for carbon tetrachloride.

TABLE 1 REQUIREMENTS FOR CARBON TETRACHLORIDE

(Clause 3.2)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Appendix A)
		Pure Grade	Technical Grade	
(1)	(2)	(3)	(4)	(5)
i)	Relative density* at 27°C/27°C	1·584 to 1·590†	1·580 to 1·592†	A-2
ii)	Distillation range at 760 mm Hg	2 to 97 ml shall distil between 76·0 to 77·0°C	2 to 97 ml shall distil between 75·8 to 77·2°C	A-3
iii)	Non-volatile matter, percent by mass, *Max*	0·002	0·01	A-4
iv)	Moisture content, ppm (by mass), *Max*	50	—	A-5
v)	Acidity or alkalinity	Neutral	Neutral	A-6
vi)	Spot test	To pass test	To pass test	A-7
vii)	Sulphur compounds (as CS_2), percent by mass, *Max*	0·001	0·1	A-8
viii)	Free chlorine	To pass test	To pass test	A-9
ix)	Methyl chloride, methylene chloride and chloroform, percent by mass, *Max*	0·025	—	A-10
x)	Colour in Hazen units, *Max*	15	15	A-11

*Relative density is the term adopted for specific gravity with water as reference substance by the International Organization for Standardization (ISO).

†The relative density determined at any temperature within the range 25 to 35°C can be adjusted to 27°/27°C using the correction factor of + 0·001 6 for every degree Celsius fall and — 0·001 6 for every degree Celsius rise in temperature.

5.2.2 All containers in which fire extinguishing blend is stored or transported shall be prominently and clearly marked with the following:

'VOLATILE, POISONOUS SOLVENT. USE WITH ADEQUATE VENTILATION. AVOID PROLONGED BREATHING OF VAPOUR. POISONOUS GASES FORMED WHEN USED TO EXTINGUISH FLAME OR ON CONTACT WITH HOT SURFACES.'

5.2.3 The containers may also be marked with the Standard Mark.

5.2.4 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6. SAMPLING

6.1 Representative samples of the material shall be drawn and their conformity to this standard judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR CARBON TETRACHLORIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 Outline of the Method — In this method, mass of equal volumes of the material and water at the same temperatures are compared using relative density bottle.

A-2.1 Apparatus

A-2.1.1 *Relative Density Bottle* — with well-fitting ground-glass joints. To calibrate, clean and dry the bottle thoroughly, weigh and then fill with water, freshly boiled and cooled to 27°C, after removing the cap. Fill to overflowing by holding the bottle on its side in such a manner as to prevent entrapment of air bubbles, insert the stopper and immerse in a water-bath at 27·0 ± 0·2°C. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes.

*Specification for water for general laboratory use (*second revision*).

Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to a lower temperature and weigh. Calculate the mass of water. The relative density bottle of about 50 ml capacity and of either of the two shapes shown in Fig. 1 is recommended.

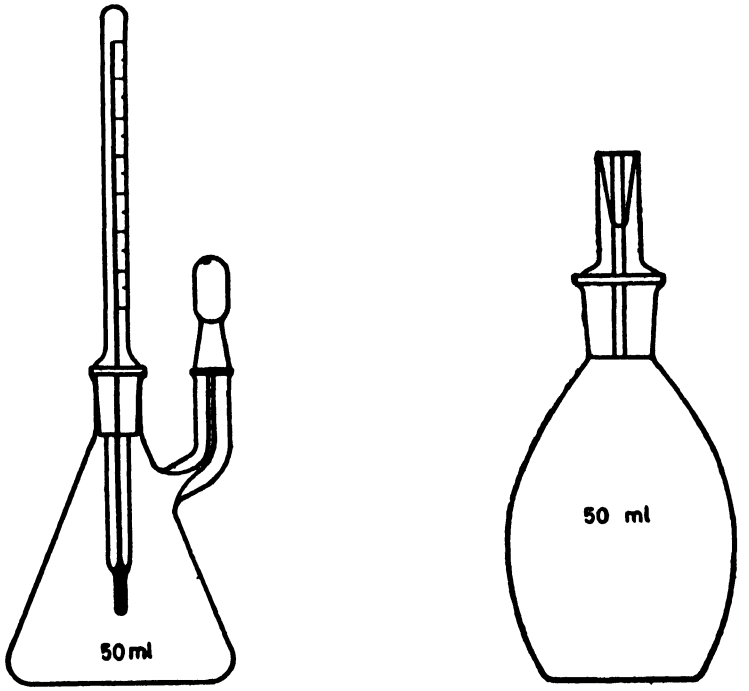


FIG. 1 RELATIVE DENSITY BOTTLES

A-2.1.2 *Water-Bath* — maintained at 27·0 ± 0·2°C.

A-2.1.3 *Thermometer* — any convenient thermometer of a suitable range with 0·1 or 0·2 deg subdivisions.

A-2.1.3.1 The thermometer shall be checked against a standard thermometer, which has been calibrated and certified by the National Physical Laboratory, New Delhi, or any other organization recognized for such work.

A-2.2 Procedure — Fill the relative density bottle with the material previously kept at about 27°C to overflowing, holding the bottle on its side in such a manner as to prevent entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water-bath maintained at 27·0 ± 0·2°C and hold for 30 minutes. Carefully wipe off any liquid which has come through the capillary opening. Remove the relative density bottle from the bath, clean, dry it thoroughly, cool and weigh.

A-2.3 Calculation

$$\text{Relative density at } 27°C/27°C = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle filled with the material at 27°C,

B = mass in g of the relative density bottle, and

C = mass in g of the relative density bottle filled with water at 27°C.

NOTE — The relative density determined at any temperature within the range 25 to 35°C can be adjusted to 27°C/27°C using the correction factor of + 0·001 6 for every degree Celsius fall, and −0·001 6 for every degree Celsius rise in temperature.

A-3. DETERMINATION OF DISTILLATION RANGE

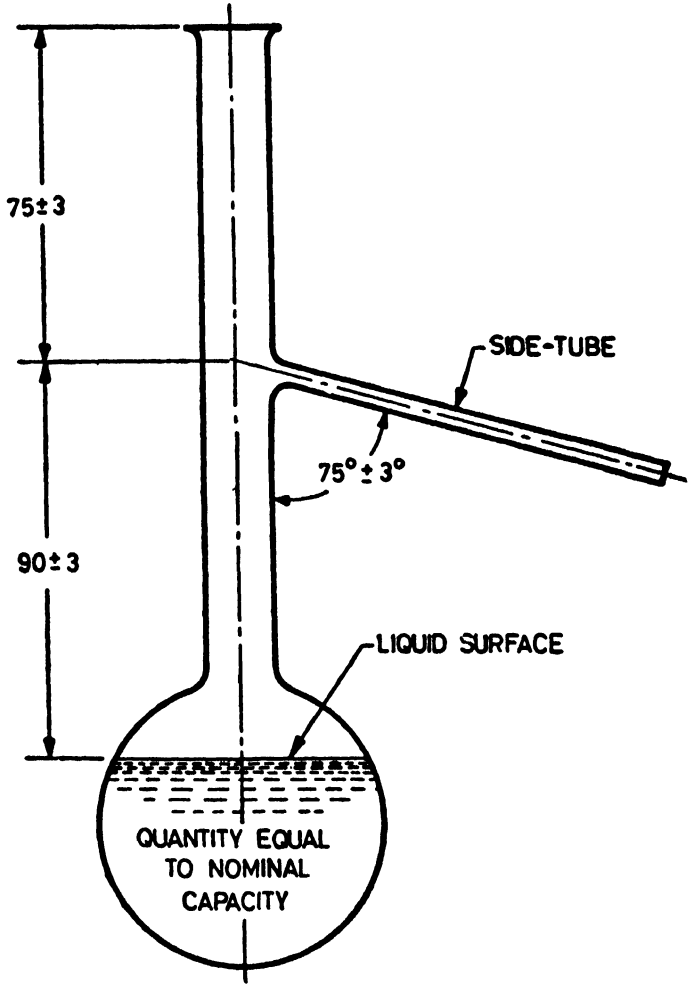
A-3.0 Outline of the Method — The material is distilled in the specified apparatus under the prescribed condition of heat input and rate of distillation. One hundred millilitres of the material are distilled and percentages by volume of the distillate recovered at the specified temperatures are recorded.

A-3.1 Apparatus

A-3.1.1 *Distillation Flask* — of the shape and dimensions shown in Fig. 2.

A-3.1.2 *Thermometer* — a suitable partial immersion thermometer having a range of 48 to 100°C, graduated at each 0·2 deg and with a maximum scale error of 0·2 deg. The maximum overall length of the thermometer shall be 385 mm and stem diameter shall be 5·5 to 8·0 mm.

A-3.1.2.1 The thermometer shall bear a certificate of the National Physical Laboratory, New Delhi, or any other organization recognized for such work.

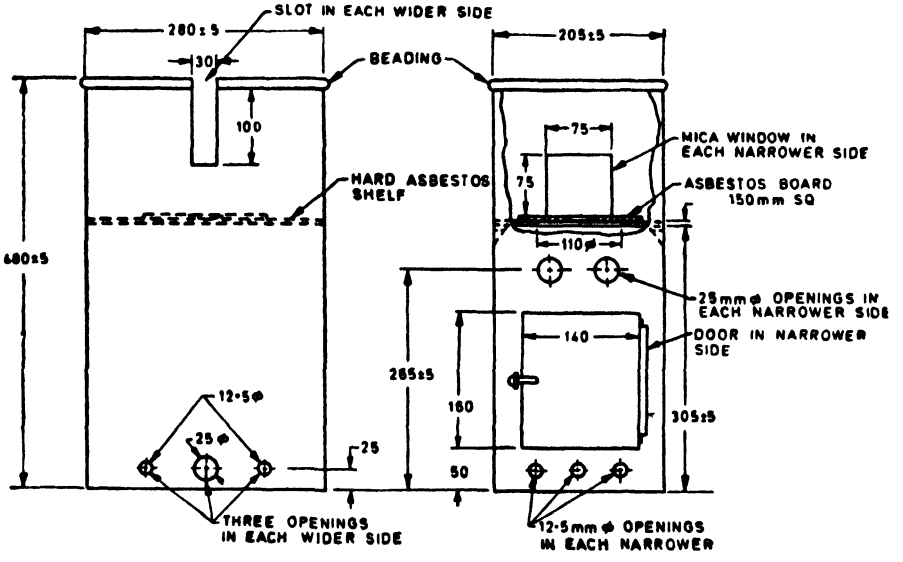


All dimensions in millimetres.

FIG. 2 DISTILLATION FLASK

A-3.1.3 *Rectangular Draught Screen* — rectangular in cross-section, made of 0·8 mm thick sheet metal, with the dimensions shown in Fig. 3 and open at the top and bottom. It shall comply with the following requirements:

a) In each of the two narrower sides of the draught screen there shall be two circular holes, each 25 mm diameter, and in each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 3. The diameter of each of the holes centrally situated in the longer sides shall be 25 mm and of the remaining ten holes shall be 12·5 mm. At the middle of each of the wider sides a vertical slot with the dimensions shown in Fig. 3 shall be cut downwards from the top of the screen. A removable shutter conforming to the dimensions shown in Fig. 4 shall be provided for closing the vertical slot not in use.



All dimensions in millimetres.

FIG. 3 RECTANGULAR DRAUGHT SCREEN

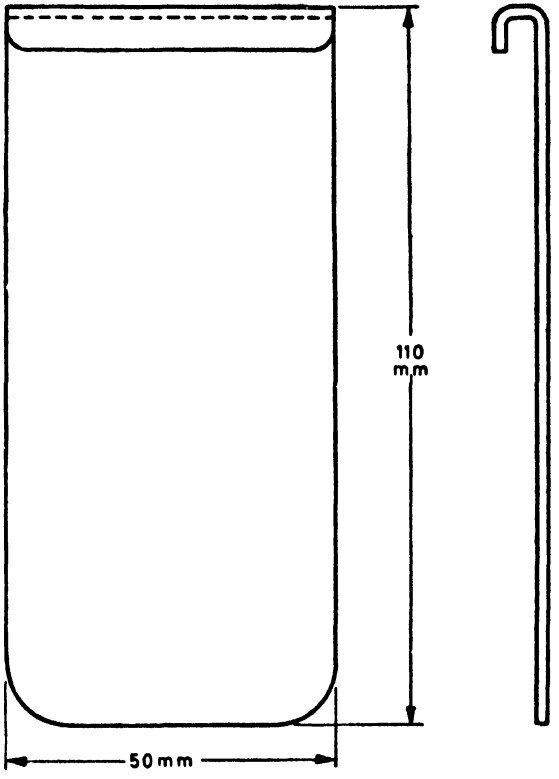


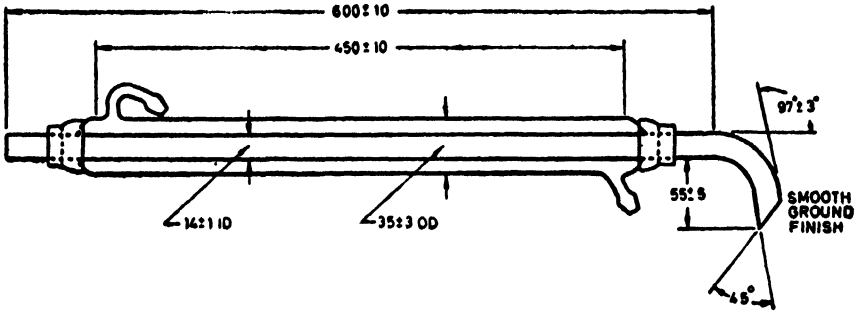
FIG. 4 REMOVABLE SHUTTER

b) A shelf of hard asbestos board, 6 mm in thickness and having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

c) In one of the narrower sides of the screen a door shall be provided having dimensions and position as shown in Fig. 3. In each of the narrower sides of the screen a mica window shall be placed centrally with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 3.

d) An asbestos board 150 × 150 × 6 mm in size having a central hole 50 mm in diameter shall be so placed on the asbestos shelf that the two holes are approximately concentric and the distillation flask when in position completely closes the hole of the asbestos board.

A-3.1.4 *Liebig Condenser* — With the bent end made of Type I glass (graded according to IS : 2303-1963*), with a wall thickness of 1·0 to 1·5 mm and conforming to the shape and dimensions shown in Fig. 5. The bent portion may, however, be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.



All dimensions in millimetres.

FIG. 5 LIEBIG CONDENSER

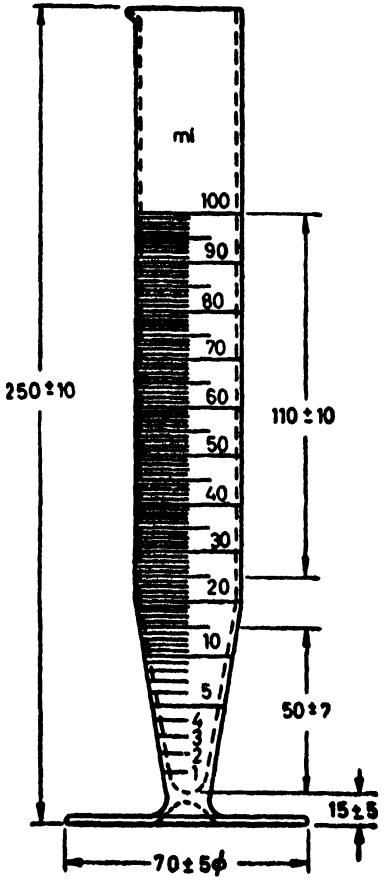
A-3.1.5 *Receiver* — of 100-ml capacity, with dimensions and graduation as shown in Fig. 6.

A-3.1.6 *Electric Heater, Gas Burner or Other Flame Type Heater* — any suitable heater or burner that enables the distillation to be carried out as described in **A-3.2**.

A-3.2 Procedure — Assemble the apparatus as shown in Fig. 7. Measure 100 ml of the material at laboratory temperature by means of the receiver and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Fit the thermometer in the flask so that the bottom of the capillary is in level with the lower edge of the side-tube joint. Pass an adequate supply of cooling water at 0 to 5°C through the condenser. To receive

*Method of grading glass for alkalinity.

the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly; especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be not less than 5 nor greater than 10 minutes. Place the receiver in a manner that the condensate flows down its side. Continue the distillation at the rate of 4 to 5 ml per minute (about 2 drops per second). Read the temperature on the thermometer when 2 ml and 97 ml of the distillate have been collected in the cylinder.



All dimensions in millimetres.

FIG. 6 RECEIVER

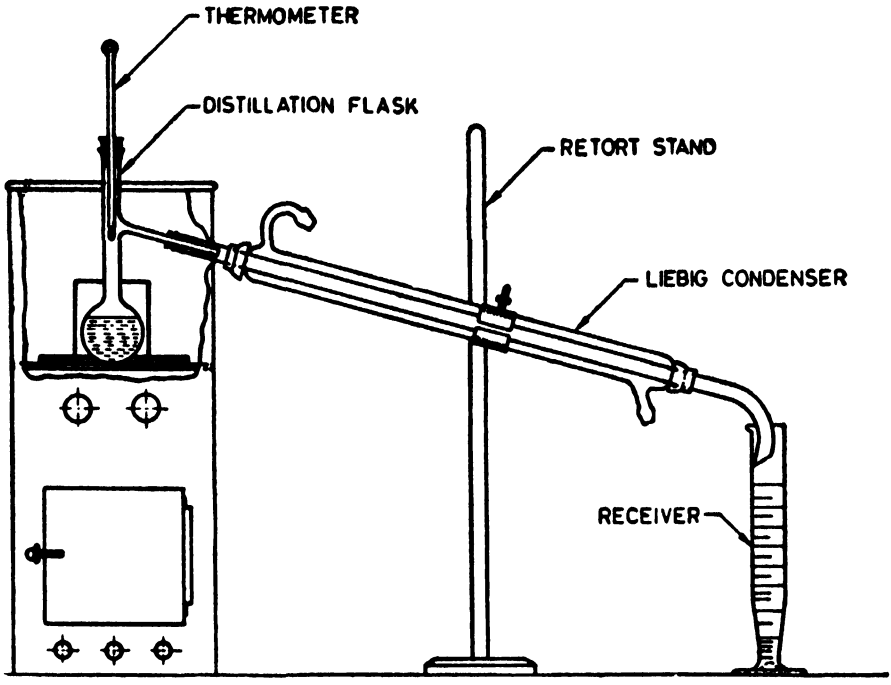


FIG. 7 ASSEMBLY OF APPARATUS FOR DETERMINING DISTILLATION RANGE

A-3.3 Correction of the Thermometer Reading

A-3.3.1 *Error of Scale* — In all the thermometer readings, make the corrections as indicated on the certificate of the instrument.

A-3.3.2 *Correction for Barometric Pressure* — If the barometric pressure prevailing during the determination is normal, namely, 760 mm Hg, no correction need be applied to the specified temperature and the thermometer scale as corrected under **A-3.3.1** shall be used as such. If, however, the prevailing pressure deviates from 760 mm Hg, the specified temperatures shall also be corrected as follows:

a) For every 10 mm Hg above 760 mm Hg, subtract 0·43 deg from the specified temperature; and

b) For every 10 mm Hg below 760 mm Hg, add 0·43 deg to the specified temperature.

NOTE — These corrections are valid only for pressures above 700 mm Hg.

A-4. DETERMINATION OF NON-VOLATILE MATTER

A-4.0 Outline of the Method — A known quantity of the material is evaporated on a water-bath and the residue is weighed.

A-4.1 Apparatus

A-4.1.1 *Basin* — of suitable capacity; made of platinum, silica or borosilicate glass.

A-4.2 Procedure — Evaporate 100 ml of the material to dryness in the weighed basin on a water-bath. Dry the residue for 30 minutes in an oven maintained at a temperature of 100 ± 2°C. Cool in a desiccator and weigh.

A-4.3 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{M}{d}$$

where

M = mass in g of the residue obtained, and

d = relative density of the material.

A-5. DETERMINATION OF MOISTURE CONTENT

A-5.1 Take 100 ml of the material and determine its moisture content as prescribed in IS : 2362-1973*.

A-6. TEST FOR ACIDITY OR ALKALINITY

A-6.1 Prepared Aqueous Extract — Shake 25 ml of the material with 25 ml of freshly boiled and cooled water for three minutes and allow the layers to separate. Reserve the aqueous layer for the test.

A-6.2 Reagents

A-6.2.1 *Methyl Orange Indicator* — Dissolve 0·50 g of the methyl orange in water and dilute to one litre.

A-6.2.2 *Bromocresol Purple Solution* — Warm 0·1 g of bromocresol purple with 5 ml of rectified spirit conforming to IS : 323-1959†. Dilute to 100 ml with 20 percent ethyl alcohol, add 3·7 ml of 0·05 N sodium hydroxide solution and dilute to 250 ml with 20 percent ethyl alcohol.

*Determination of water by the Karl Fischer method (*first revision*).
†Specification for rectified spirit (*revised*).

A-6.3 Procedure — Take 10 ml of the *prepared aqueous extract* (*see* **A-6.1**) and add 4 drops of methyl orange indicator. Carry out a control test by adding the same amount of indicator to 10 ml of freshly boiled and cooled distilled water, and compare the colour produced in both. Repeat this test using 4 drops of bromocresol purple solution in place of methyl orange indicator.

A-6.4 The material shall be taken as neutral if the colour obtained with the *prepared aqueous extract* shows no difference when compared with the colour produced in the control test.

A-7. SPOT TEST

A-7.1 Procedure — Place 5 drops of the material near the centre of a filter paper (Whatman No. 40 or equivalent), taking care that the wet portion of the filter paper does not come into contact with any object. Allow to evaporate in a well-ventilated room and keep for two hours.

A-7.2 The material shall be taken to have satisfied the test if no spot or stain is left on the filter paper.

A-8. DETERMINATION OF SULPHUR COMPOUNDS (AS CS_2)

A-8.0 Principle — Carbon disulphide present in the material reacts with cupric ions and diethylamine to form copper diethyl dithio carbamate. This compound is soluble in carbon tetrachloride giving a yellow colour, the intensity of which is a function of the carbon disulphide content. Intensity of the colour is determined photometrically at about 425 nm.

A-8.1 Reagents

A-8.1.1 *Colour Reagent* — Place 50 ml of water in a 1 000 ml one-mark volumetric flask, add 0·060 g of cupric acetate monohydrate [$Cu(CH_3COO)_2.H_2O$] and shake until dissolution is complete. Add 10·0 ml of diethylamine and 20·0 ml of triethanolamine. Dilute to the mark with 95 percent (*v/v*) ethanol and mix. Store the solution in the dark, in a closed flask, preferably closed by means of a polyethylene stopper.

A-8.1.2 *Carbon Tetrachloride* — free from carbon disulphide.

A-8.1.3 *Carbon Disulphide*

A-8.1.4 *Standard Carbon Disulphide Solution* — Introduce about 50 ml of carbon tetrachloride into a 100-ml one-mark volumetric flask and add 10·0 ml of carbon disulphide. Dilute to the mark with carbon tetrachloride and mix.

The solution thus obtained has a concentration of 8·09 percent (*m/m*) of carbon disulphide in carbon tetrachloride (solution A).

A-8.1.4.1 Take 10·0 ml of *solution A* and transfer to a 100-ml one-mark volumetric flask. Dilute to the mark with the carbon tetrachloride and mix. The *solution B* thus obtained has a concentration of 0·79 percent (*m/m*) of carbon disulphide in carbon tetrachloride. Repeat the operation twice. The *solution C* finally obtained has a concentration of 0·007 9 percent (*m/m*) of carbon disulphide in carbon tetrachloride.

A-8.2 Apparatus

A-8.2.1 *Spectrophotometer or Photoelectric Absorptiometer* — fitted with appropriate filters.

A-8.2.2 *Chronometer* — for measuring time up to 5 minutes.

A-8.3 Procedure

A-8.3.1 *Preparation of Calibration Curve*

A-8.3.1.1 *Preparation of standard matching solution* — into a series of four 100-ml one-mark volumetric flasks, place respectively 5·0, 7·5, 10·0, 12·5 ml of the standard carbon disulphide *solution C*. Dilute to the mark with the carbon tetrachloride and mix. The standard matching solutions thus prepared contain respectively 0·000 4 g, 0·000 6 g, 0·000 8 g and 0·001 0 g of carbon disulphide (CS_2) in carbon tetrachloride.

A-8.3.2 *Development of Colour* — By means of a pipette, place 50 ml of the colour reagent into each of two 100-ml conical flasks. To one of the flasks add, by means of a pipette, 5·0 ml of the standard matching solution and to the other 5 0 ml of the carbon tetrachloride (compensating solution). Start the chronometer, mix and transfer the solution to the two cells of the spectrophotometer with appropriate optical path length.

A-8.3.3 *Photometric Measurement* — Carry out the measurements for 5 minutes after the start of colour development using the spectrophotometer at a wavelength in the region of 425 nm or the photoelectric absorptiometer fitted with appropriate filters, adjusting the instrument to zero absorbance against the compensating solution. Carry out the operations of colour development and photometric measurement for each of the standard matching solutions.

A-8.3.4 *Preparation of Calibration Chart* — Prepare a calibration chart having, the carbon disulphide contents as percentages by mass as abscissae and the corresponding values of the photometric measurements as ordinates.

A-8.4 Determination

A-8.4.1 *Development of Colour* — By means of a pipette, place 50 ml of the colour reagent in two 100-ml conical flasks. To one of the flasks add, by means of a pipette, 5·0 ml of the sample and to the other 5·0 ml of the carbon tetrachloride (compensating solution). Start the chronometer, mix and transfer the solutions to the cells of the apparatus.

A-8.4.2 *Photometric Measurements* — Carry out the photometric measurements for 5 minutes after the start of the colour development following the directions given in **A-8.3.3**. Determine, by means of the calibration chart the carbon disulphide (CS_2) content, expressed as a percent by mass. If the sample is diluted with carbon tetrachloride, take this dilution into account in calculating the results.

NOTE — For technical grade of the material dilute 10 ml of the sample to 1 000 ml using carbon tetrachloride and prepare the standard matching solution (*see* **A-8.3.1.1**). Proceed for the determination of carbon disulphide as above. Take account of the dilution while calculating the results.

A-9. TEST FOR FREE CHLORINE

A-9.0 Outline of the Method — The material is shaken with 3,3'-dimethylnaphthidine solution and the colour developed, if any, is noted.

A-9.1 Apparatus

A-9.1.1 *Graduated Measuring Cylinder* — 50-ml capacity, glass stoppered (*see* IS : 878-1956*).

A-9.2 Reagent

A-9.2.1 *3,3'-dimethylnaphthidine Solution* — Dissolve 0·01 g of finely ground 3,3'-dimethylnaphthidine in 5 ml of glacial acetic acid and dilute rapidly with water to 200 ml. Store the solution in the dark.

A-9.3 Procedure — To 50 ml of the material contained in the graduated measuring cylinder, add 5 ml of the 3,3'-dimethylnaphthidine solution and shake the cylinder for 30 seconds.

NOTE — The test shall be carried out in the dark and colour developments shall be checked immediately.

A-9.4 The material shall be regarded to have passed the test if no pink colour is developed.

*Specification for graduated measuring cylinders.

A-10. DETERMINATION OF METHYL CHLORIDE, METHYLENE CHLORIDE AND CHLOROFORM

A-10.1 Apparatus

A-10.1.1 *Gas Chromatograph* — sample as well as reference columns, two number, both 550 silicone, 20 percent on chromosorb W (60 to 80 mesh) 6 mm stainless steel tubing/copper tubing, 3·6 metres in length. It shall be equipped with thermal conductivity detector.

A-10.1.2 *Microlitre Syringe with Fixed Needle* — 5 cm long, 50 μl capacity.

A-10.1.3 *Recorder* — minus 0·05 to plus 1·05 mV, 25 cm chart, response one second full scale, and chart speed of 25 mm per two minutes.

A-10.2 Reagents

A-10.2.1 *Acetone*

A-10.2.2 *Synthetic Standard of Pure Carbon Tetrachloride* — containing known amounts of methyl chloride, methylene chloride and chloroform.

A-10.3 Procedure

A-10.3.1 Helium or hydrogen may be used as the carrier gas. The operating parameters of the gas chromatograph when using helium or hydrogen shall be as follows:

	Helium	*Hydrogen*
a) Detector block temperature	265°C	265°C
b) Injection port temperature	185°C	185°C
c) Column oven temperature	80°C	80°C
d) Bridge current	200 mA	200 mA
e) Carrier gas pressure	50 psig	20 psig
f) Flow-rate	40-70 ml/min	40-70 ml/min

A-10.3.2 *Sample Injection* — Rinse the 50 μl syringe with acetone twice and dry for two minutes or longer. Rinse the syringe twice before filling it to the 20 μl mark. Inject the standard solution into the gas chromatograph and obtain a standard chart. Rinse the syringe twice with the material under test and inject 20 μl into the chromatograph. It is imperative that the same injection technique is used and the syringe used in this operation is not used with any other type of solvent, which is very necessary to prevent contamination.

A-10.3.3 The components will elute in the order of their boiling points:

First methyl chloride
Second methylene chloride
Third chloroform
Fourth carbon tetrachloride

A-10.4 Calculation — Using a rule marked in fifty 0·02 units to the 25 mm determine the peak height of each component. Refer to the calibration curves for the concentration of component present.

A-11. TEST FOR COLOUR

A-11.0 Outline of the Method — The colour of the material is compared with that of the colour standard and expressed in terms of Hazen colour units. The Hazen colour which is defined as the colour of an aqueous solution, containing 1 part per million of platinum in the form of chloroplatinic acid and 2 parts per million of cobaltous chloride ($CoCl_2.6H_2O$).

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinders* — two, 100-ml capacity (*see* IS : 4161-1967*).

A-11.1.2 *One-Mark Graduated Flasks* — 250-ml and 500-ml capacities (*see* IS : 915-1958†).

A-11.2 Reagents

A-11.2.1 *Cobaltous Chloride Hexahydrate* — solid.

A-11.2.2 *Hydrochloric Acid* — conforming to IS : 265-1962‡.

A-11.2.3 *Chloroplatinic Acid* — Dissolve 250 mg of platinum in small quantity of aquaregia contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

A-11.3 Preparation of Colour Standard — Dissolve 0·50 g of the cobaltous chloride hexahydrate and whole of the chloroplatinic acid in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500 ml one-mark graduated flask. Dilute with water to the mark.

*Specification for Nessler cylinders.
†Specification for one-mark graduated flasks.
‡Specification for hydrochloric acid (*revised*).

A-11.3.1 Pipette 7·5 ml of the solution (*see* **A-11.3**) into a 250 ml one-mark graduated flask and dilute with water to the mark. The diluted solution corresponds to a colour of 15 Hazen units and should always be freshly prepared.

A-11.4 Procedure — Fill one of the Nessler cylinders to the mark with the material to be tested, and the other with the colour standard. Compare the colours using a white background.

A-11.4.1 The material shall be taken to have passed the test if the colour of the sample is not darker than that of the colour standard.

APPENDIX B

(*Clause* 6.1)

SAMPLING OF CARBON TETRACHLORIDE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampling instrument shall be clean and dry.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both by suitable means, or by rolling.

B-1.4 The samples shall be placed in suitable, clean, dry and airtight, metal, or dark or amber glass containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

A-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

B-1.7 Samples shall be stored in the dark.

B-2. SAMPLING INSTRUMENT

B-2.1 The following sampling instrument may be used:

a) Sampling bottle or can, for taking samples from tanks or drums; and

b) Sampling tube, for taking samples from bottles or small containers.

B-2.1.1 *Sampling Bottle or Can* — consists of a weighed glass or metal containers with removable stopper or top to which is attached a light chain (*see* Fig. 8). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

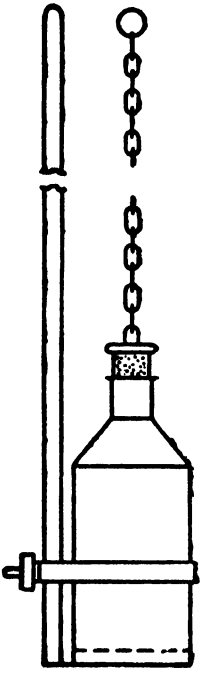


FIG. 8 SAMPLING BOTTLE

B-2.1.2 *Sampling Tube* — made of metal or thick glass, 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 9). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

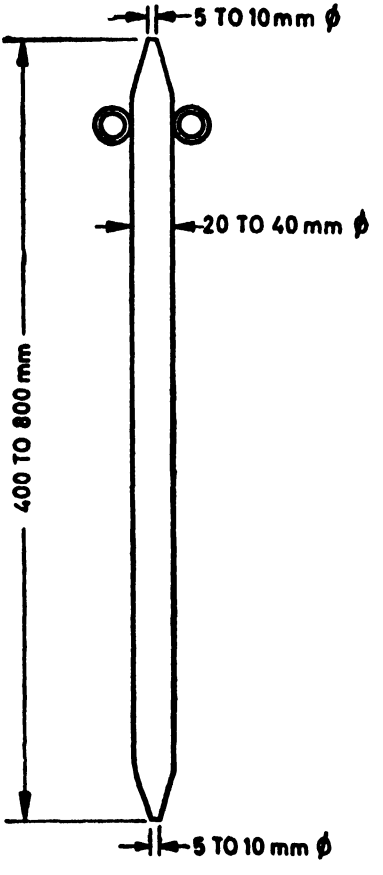


FIG. 9 SAMPLING TUBE

B-2.1.2.1 For small containers the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 For Tanks and Drums — Each tank or drum shall be sampled separately.

B-3.2 For Bottles and Small Containers — Each lot (*see* **B-3.2.1**) shall be sampled separately.

B-3.2.1 *Lot* — In any consignment, all the containers of the same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different grades, the containers belonging to the same batch and grade shall be grouped together and each group shall constitute a separate lot.

B-3.2.2 The number of containers (n) to be selected from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

Lot Size	No. of Containers to be Selected
N	n
(1)	(2)
Up to 15	3
16 ,, 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

B-3.2.3 These containers shall be selected from the lot at random. In order to ensure the randomness of selection, a random number table may be used. For guidance and use of random number table, IS : 4905-1968* may be referred. In the absence of random number table, the following procedure may be adopted:

Starting from any container in the lot, count the containers as 1, 2, 3,....., etc, up to r and so on, in one order. Every rth container thus counted shall be withdrawn to constitute the sample where r is the integral part of N/n (N and n being the lot size and sample size respectively).

B-4. TEST SAMPLE AND REFEREE SAMPLE

B-4.1 From Tanks and Drums — As far as possible, samples from a tank or drum should be drawn during the operation of filling. In that case, equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material collected shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

*Methods for random sampling.

B-4.2 From Bottles and Small Containers — From each of the bottles or containers selected according to **B-3.2.3**, a small representative portion of the material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 500 ml. This shall be divided into three equal parts, one for the purchaser another for the supplier and the third for the referee.

B-4.3 All the test samples shall be transferred to separate sample containers and sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

B-5. TESTS

B-5.1 Tests for the determination of all the requirements given in this specification shall be performed on the composite sample obtained as in **B-4.1** or **B-4.2**.

B-6. CRITERIA FOR CONFORMITY

B-6.1 The lot shall be declared as conforming to this specification if all the test results satisfy the prescribed requirements.

BUREAU OF INDIAN STANDARDS

Headquarters:
Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402
Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

	Telephone
Central Laboratory:	
Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010	8-77 00 32
Regional Offices:	
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9 Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95
Branch Offices:	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G. T. Road, GHAZIABAD 201001	8-71 19 96
53/5 Ward No. 29, R. G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-58C, L. N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
Patliputra Industrial Estate, PATNA 800013	26 23 05
T. C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	32 36 35

*Sales Office is at 5 Chowringhee Approach, P. O. Princep Street, CALCUTTA 700072 — 27 10 85

†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007 — 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002 — 222 39 71

Printed at New India Printing Press, Khurja, India